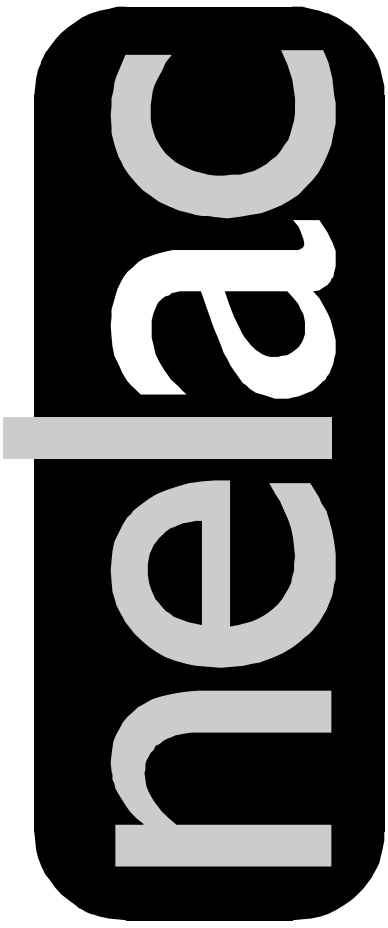
National Environmental Laboratory **Accreditation** Conference



QUALITY SYSTEMS

Abstract of

PROPOSED CHANGES

May 15, 2002

Proposed Changes to NELAC Chapter 5
Quality Systems
May 1, 2002

Section 5.1 SCOPE

d) This Standard includes data integrity procedures that provide assurance that a highly ethical approach to testing is a key component of all laboratory planning, training and implementation of methods. The following sections in this standard address data integrity procedures:

Quality Manual	5.5.2.u
Internal Audits	5.5.3.1.1
Data Integrity	5.5.3.6
Personnel Training	5.6.2.h

Section 5.5.2 Quality Manual

The quality manual and related quality documentation shall also contain:

~~u) ethics policy statement developed by the laboratory and processes/procedures for educating and training personnel in their ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions;~~

u) data integrity procedures defined in detail within the quality manual and having these four required elements: 1) data integrity training, 2) signed data integrity documentation for all laboratory employees, 3) in-depth, periodic monitoring of data integrity and 4) data integrity procedure documentation;

Section 5.5.3 Audits, Reviews, ~~and~~ Corrective Actions and Data Integrity

Section 5.5.3.1 Internal Audits

5.5.3.1.1 Data Integrity Audits

The laboratory, as part of their internal auditing program, shall insure that a review is conducted with respect to any evidence of inappropriate actions or vulnerabilities related to data integrity. Discovery of potential issues shall be handled in a confidential manner until such time as a follow up evaluation, full investigation, or other appropriate actions have been completed and the issues clarified. All investigations that result in finding of inappropriate activity shall be documented and shall include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients. All documentation of these investigation and actions taken shall be maintained for at least five years.

Section 5.5.3.6 Data Integrity

5.5.3.6 Data Integrity

Laboratory management shall provide a mechanism for confidential reporting of data integrity issues in their laboratory. A primary element of the mechanism is to assure confidentiality and a receptive environment in which all employees may privately discuss ethical issues or report items of ethical concern. In instances of ethical concern, the mechanism shall include a process

whereby Laboratory management are to be informed of the need for any further detailed investigation.

The data integrity procedures shall be signed and dated by senior management. These procedures and the associated implementation records shall be properly maintained and made available for assessor review. The data integrity procedures shall be annually reviewed and updated by management.

Section 5.6.2 Laboratory Management Responsibilities

In addition to 5.4.2.d, the laboratory management shall be responsible for:

~~h) Developing a proactive program for prevention and detection of improper, unethical or illegal actions. Components of this program could include: internal proficiency testing (single and double blind); post-analysis, electronic data and magnetic tape audits; effective reward program to improve employee vigilance and co-monitoring; and separate SOPs identifying appropriate and inappropriate laboratory and instrument manipulation practices.~~

h) Data integrity training shall be provided as a formal part of new employee orientation and must also be provided on an annual basis for all current employees. Topics covered shall be documented in writing and provided to all trainees. Key topics covered during training must include organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting, how and when to report data integrity issues, and record keeping. Training shall include discussion regarding all data integrity procedures, data integrity training documentation, in-depth data monitoring and data integrity procedure documentation. Employees are required to understand that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences including immediate termination, debarment or civil/criminal prosecution. The initial data integrity training and the annual refresher training shall have a signature attendance sheet or other form of documentation that demonstrates all staff have participated and understand their obligations related to data integrity. Senior managers acknowledge their support of these procedures by 1) upholding the spirit and intent of the organization's data integrity procedures and 2) effectively implementing the specific requirements of the procedures.

Specific examples of breaches of ethical behavior should be discussed including improper data manipulations, adjustments of instrument time clocks, and inappropriate changes in concentrations of standards. Data integrity training requires emphasis on the importance of proper written narration on the part of the analyst with respect to those cases where analytical data may be useful, but are in one sense or another partially deficient. The data integrity procedures may also include written ethics agreements, examples of improper practices, examples of improper chromatographic manipulations, requirements for external ethics program training, and any external resources available to employees.

Section 5.9.4.2 Instrument Calibration

~~**Note: In the following sections, initial instrument calibration is directly used for quantitation and continuing instrument calibration verification is used to confirm the continued validity of the initial calibration.**~~

Section 5.9.4.2.1 Initial Instrument Calibration

~~c) Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.~~

~~d~~c) All initial instrument calibrations must be verified with a standard obtained from a second manufacturer or lot if the lot can be demonstrated from the manufacturer as prepared independently from other lots. Traceability shall be to a national standard, when available.

~~e~~d) Criteria for the acceptance of an initial instrument calibration must be established, e.g., correlation coefficient or relative percent difference. The criteria used must be appropriate to the calibration technique employed.

~~f~~e) Results of samples not bracketed by initial instrument calibration standards (within calibration range) must be reported ~~as having less certainty, e.g.,~~ with defined qualifiers or flags or explained in the case narrative. The lowest calibration standard must be above the detection limit. Noted exception: The following shall occur for instrument technology (such as ICP or ICP/MS) with validated techniques from manufacturers or methods employing standardization with a zero point and a single point calibration standard: Standardization of the instruments using the zero point and single standard shall be performed with each analytical batch. Once the instrument is standardized with the zero point and the single standard, the linear working range must then be defined by the analysis of a series of reference standards, one of which must be at the minimum quantitation limit. Once the linear range is established it shall be routinely checked at a frequency and using procedures as established by the method and/or manufacturer. The minimum quantitation limit (MQL) shall be demonstrated with each analytical batch by the analysis of a reference standard at a concentration corresponding to the MQL, with results meeting established acceptance criteria. If an individual sample analysis produces results above the single point calibration standard, one of the following actions shall occur: (1) analyze a reference standard at or above the sample value that meets established acceptance criteria for validating the linearity; (2) dilute the sample such that the result falls below the single point calibration concentration; (3) report the data with an appropriate data qualifier and/or explain in the case narrative.

~~g~~f) If the initial instrument calibration results are outside established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed. If for any reason reanalysis of the samples is not possible, ~~D~~data associated with an unacceptable intial instrument calibration shall not be reported without appropriate data qualifiers.

~~h~~g) Calibration standards must include concentrations at or below the regulatory limit/decision level, if these limits/levels are known by the laboratory, unless these concentrations are below the laboratory's demonstrated detection limits (See D.1.4 Detection Limits)

~~i~~h) If a reference or mandated method does not specify the number of calibration standards, the minimum number is two, not including blanks or a zero standard with the noted exception of instrument technology for which it has been established by methodologies and procedures that a zero and a single point standard are appropriate for calibrations (see 5.9.4.2.1.f). The laboratory must have a standard operating procedure for determining the number of points for establishing the initial instrument calibration.

Section 5.11.3 Sample Receipt Protocols

2) The laboratory shall implement procedures for checking chemical preservation using readily available techniques, such as pH or free chlorine, prior to or during sample preparation or analysis.

Microbiological samples from chlorinated public water systems do not require an additional chlorine residual check in the laboratory if the following conditions are met:

i. sufficient sodium thiosulfate is added to each container to neutralize at minimum 5 mg/l of chlorine;

ii. one container from each batch of laboratory prepared containers or lot of purchased ready-to-use containers is checked to ensure efficacy of the sodium thiosulfate to 5 mg/l chlorine and the check is documented;

iii. chlorine residual is checked in the field and documented on the chain of custody.

Appendices:
D.1.1 Positive and Negative Controls

b) Positive Control – Method Performance

Evaluation Criteria and Corrective Action: The results of the individual batch LCS are calculated in percent recovery or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall document the calculation ~~for percent recovery~~.

The individual LCS is compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits or utilize client specified assessment criteria.

A LCS that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with a LCS determined to be "out of control" ~~should~~ shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes.

c) Sample Specific Controls

Evaluation Criteria and Corrective Action: The results from matrix spike/matrix spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R), ~~and~~ relative percent difference (RPD) or other appropriate statistical technique that allows comparison to established acceptance criteria. The laboratory shall document the calculation for %R, relative percent difference or other statistical treatment used.

The results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory ~~should~~ shall determine internal criteria and document the method used to establish the limits. For matrix spike results outside established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.

D.3.1 Sterility Checks and Blanks, Positive and Negative Controls

a2) For ~~each filtration series in the~~ filtration technique, the laboratory shall ~~prepare at least~~ conduct one beginning and one ending sterility check for each filtration unit used in a filtration series. The filtration series may include single or multiple filtration units, which have been sterilized prior to beginning the series. ~~When an interruption of more than 30 minutes occurs, the filtration funnels shall be re-sterilized.~~ The filtration series is considered ended when more than 30 minutes elapses between successive filtrations. During a filtration series, filter funnels must be rinsed with three 20-30 ml portions of sterile rinse water after each sample filtration. In addition, laboratories must insert a sterility blank after every 10 samples or sanitize filtration units by UV light after each sample filtration.

D.3.6 Quality of Standards, Reagents and Media

a) Culture media may be prepared from commercial dehydrated powders or may be purchased ready to use. ~~Preparation from different chemical ingredients shall not be done unless the media is not available commercially or unless specified by the method.~~ Media may be prepared by the laboratory from basic ingredients when commercial media are not available or when it can be demonstrated that commercial media do not provide adequate results. Media prepared by the laboratory from basic ingredients must be tested for performance (e.g., for selectivity, sensitivity, sterility, growth promotion, growth inhibition) prior to first use. Detailed testing criteria information must be defined in either the laboratory's test methods, SOPs, Quality Manual, or similar documentation.

D.6 ASBESTOS TESTING

These standards apply to laboratories undertaking the examination of asbestos samples. These standards are organized by analytical technique including transmission electron microscopy (TEM) for the analysis of water, wastewater, air, and bulk samples; phase contrast microscopy (PCM) for analysis of workplace air; and polarized light microscopy (PLM) for analysis of bulk samples. These procedures for asbestos analysis involve sample preparation followed by detection of asbestos. If NIST SRMs specified below are unavailable, the laboratory may substitute an equivalent reference material with a certificate of analysis.

D.6.1 Negative Controls

D.6.1.1 Transmission Electron Microscopy

D.6.1.1.1 Water and Wastewater

a) Blank determinations shall be made prior to sample collection. When using polyethylene bottles, one bottle from each batch, or a minimum of one from each 24 shall be tested for background level. When using glass bottles, four bottles from each 24 shall be tested. An acceptable bottle blank level is defined as $\leq$ 0.01 MFL > 10 μm. (EPA /600/R-94/134, Method 100.2, Section 8.2)

b) A process blank sample consisting of fiber-free water shall be run before the first field sample. The quantity of water shall be $\geq$ 10 mL for a 25-mm diameter filter and $\geq$ 50 mL for a 47-mm diameter filter. (EPA /600/R-94/134, Method 100.2, Section 11.8)

D.6.1.1.2 Air

a) A blank filter shall be prepared with each set of samples. A blank filter shall be left uncovered during preparation of the sample set and a wedge from that blank filter shall be prepared alongside wedges from the sample filters. At minimum, the blank filter shall be analyzed for each 20 samples analyzed. (40 CFR Part 763, Appendix A to Subpart E (AHERA), Table 1)

b) Maximum contamination on a single blank filter shall be no more than 53 structures/mm^2. Maximum average contamination for all blank filters shall be no more than 18 structures/mm^2. (AHERA, III.F.2)

D.6.1.1.3 Solid and Hazardous Waste (Bulk)

a) Contamination checks using asbestos-free material, such as the glass fiber blank in SRM 1866 (Page C-3, NIST Handbook 150-3, August 1994) shall be performed at a frequency of 1 for every 20 samples analyzed. The detection of asbestos at a concentration exceeding 0.1% will require an investigation to detect and remove the source of the asbestos contamination.

b) The laboratory must maintain a list of non-asbestos fibers that can be confused with asbestos (Section 7.5, Page C-8, NIST Handbook 150-3, August 1994). The list must include crystallographic and/or chemical properties that disqualify each fiber being identified as asbestos (Section 2.5.5.2.1 Identification, Page 54, EPA/600/R-93/116).

c) The laboratory should have a set of reference asbestos materials from which a set of reference diffraction and X-ray spectra have been developed.

D.6.1.2 Phase Contrast Microscopy

At least two (2) field blanks (or 10% of the total samples, whichever is greater) shall be submitted for analysis with each set of samples. Field blanks shall be handled in a manner representative of actual handling of associated samples in the set with a single exception that air shall not be drawn through the blank sample. A blank cassette shall be opened for approximately thirty (30) seconds at the same time other cassettes are opened just prior to analysis. Results from field blank samples shall be used in the calculation to determine final airborne fiber concentration. The identity of blank filters should be unknown to the counter until all counts have been completed. If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.

D.6.1.3 Polarized Light Microscopy

a) Friable Materials - At least one blank slide must be prepared daily or with every 50 samples analyzed, whichever is less. This is prepared by mounting a subsample of an isotropic verified non-ACM (e.g., fiberglass in SRM 1866) in a drop of immersion oil (n_D should reflect usage of various n_D's) on a clean slide, rubbing preparation tools (forceps, dissecting needles, etc.) in the mount and placing a clean coverslip on the drop. The entire area under the coverslip must be scanned to detect any asbestos contamination. A similar check must be made after every 20 uses of each piece of homogenization equipment. An isotropic verified non-ACM must be homogenized in the clean equipment, a slide prepared with the material and the slide scanned for asbestos contamination. (This can be substituted for the blank slide mentioned in this section.)

b) Non-Friable Materials - At least one non-ACM non-friable material must be prepared and analyzed with every 20 samples analyzed. This non-ACM must go through the full preparation and analysis regimen for the type of analysis being performed.

D.6.2 Test Variability/Reproducibility

D.6.2.1 Transmission Electron Microscopy

Quality assurance analyses shall be performed regularly covering all time periods, instruments, tasks, and personnel. The selection of samples shall be random and samples of special interest may be included in the selection of samples for quality assurance analyses. When possible, the checks on personnel performance shall be executed without their prior knowledge. A disproportionate number of analyses shall not be performed prior to internal or external audits. It is recommended that a laboratory initially be at 100% quality control (all samples reanalyzed). The proportion of quality control samples can later be lowered gradually, as control indicates, to a minimum of 10%.

D.6.2.1.1 Water and Wastewater

All analyses must be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings. Quality assurance analyses shall not be postponed during periods of heavy workloads. The total number of QA samples and blanks must be greater than or equal to 10% of the total sample workload. Precision of analyses is related to concentration, as gleaned from interlaboratory proficiency testing. Relative standard deviations (RSD) for amphibole asbestos decreased from 50% at 0.8 MFL to 25% at 7 MFL in interlaboratory proficiency testing, while RSD for chrysotile was higher, 50% at 6 MFL.

a) Replicate – A second, independent analysis shall be performed on the same grids but on different grid openings than used in the original analysis of a sample. Results shall be within 1.5X of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (EPA /600/R-94/134, Method 100.2, Table 2)

b) Duplicate – A second aliquot of sample shall be filtered through a second filter, prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0X of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (EPA /600/R-94/134, Method 100.2, Table 2)

c) Verified Analyses – A second, independent analysis shall be performed on the same grids and grid openings used in the original analysis of a sample. The two sets of results shall be compared according to Turner and Steel (NISTIR 5351). This shall be performed at a frequency of 1 per 20 samples. Qualified analysts must maintain an average of $\geq$ 80% true positives, $\leq$ 20% false negatives, and $\leq$ 10% false positives.

D.6.2.1.2 Air

All analyses must be performed on relocator grids so that other laboratories can easily repeat analyses on the same grid openings.

The laboratory and TEM analysts must obtain mean analytical results on NIST SRM 1876b so that trimmed mean values fall within 80% of the lower limit and 110% of the upper limit of the 95% confidence limits as published on the certificate. These limits are derived from the allowable false positives and false negatives given in Section D.6.2.1.2c, Verified Analysis, below. SRM 1876b shall be analyzed a minimum of once per year by each TEM analyst.

The laboratory must have documentation demonstrating that TEM analysts correctly classify at least 90% of both bundles and single fibrils of asbestos structures greater than or equal to 1 μm in length in known standard materials traceable to NIST, such as NIST bulk asbestos SRM 1866.

Interlaboratory analyses shall be performed to detect laboratory bias. The frequency of interlaboratory verified analysis must correspond to a minimum of 1 per 200 grid square analyses for clients.

If more than 1 TEM is used for asbestos analysis, intermicroscope analyses must be performed to detect instrument bias.

a) Replicate – A second, independent analysis shall be performed in accordance with Section D.6.2.1.1.a. (AHERA, Table III)

b) Duplicate – A second wedge from a sample filter shall be prepared and analyzed in the same manner as the original preparation of that sample. Results shall be within 2.0X of Poisson standard deviation. This shall be performed at a frequency of 1 per 100 samples. (AHERA, Table III)

c) Verified Analyses – A second, independent analysis shall be performed on the same grids and grid openings in accordance with Section D.6.2.1.1.c. (AHERA, Table III)

D.6.2.1.3 Solid and Hazardous Waste (Bulk)

Determination of precision and accuracy should follow guidelines in NISTIR 5951, Guide for Quality Control on the Qualitative and Quantitative Analysis of Bulk Asbestos Samples: Version 1. Because bulk samples with low (< 10%) asbestos content are the most problematic, a laboratory's quality control program should focus on such samples. At least 30% of a laboratory's QC analyses shall be performed on samples containing from 1% to 10% asbestos.

a) Intra-Analyst Precision - At least 1 out of 50 samples must be reanalyzed by the same analyst. For single analyst laboratories, at least 1 out of every 10 samples must be reanalyzed by the same analyst.

b) Inter-Analyst Precision - At least 1 out of 15 samples must be reanalyzed by another analyst. Inter-analyst results will require additional reanalysis, possibly including another analyst, to resolve discrepancies when classification (ACM vs. non-ACM) errors occur, when asbestos identification errors occur, or when inter-analyst precision is found to be unacceptable.

c) Inter-Laboratory Precision - The laboratory must participate in round robin testing with at least one other laboratory. Samples must be sent to this other lab at least four times per year. These samples must be samples previously analyzed as QC samples. Results of these analyses must be assessed in accordance with QC requirements. As a minimum, the QC requirements must address misclassifications (false positives, false negatives) and misidentification of asbestos types.

D.6.2.2 Phase Contrast Microscopy

a) Inter-Laboratory Precision – Each laboratory analyzing air samples for compliance determination shall implement an inter-laboratory quality assurance program that as a minimum includes participation of at least two (2) other independent laboratories. Each laboratory shall participate in round robin testing at least once every six (6) months with at least all the other laboratories in its inter-laboratory quality assurance group. Each laboratory shall submit slides typical of its own workload for use in this program. The

round robin shall be designed and results analyzed using appropriate statistical methodology. Results of this QA program shall be posted in each laboratory to keep the microscopists informed.

b) Intra- and Inter-Analyst Precision – Each analyst shall select and count a prepared slide from a "reference slide library" on each day on which air counts are performed. Reference slides shall be prepared using well-behaved samples taken from the laboratory workload. Fiber densities shall cover the entire range routinely analyzed by the laboratory. These slides shall be counted by all analysts to establish an original standard deviation and corresponding limits of acceptability. Results from the daily reference sample analysis shall be compared to the statistically derived acceptance limits using a control chart or a database. It is recommended that the labels on the reference slides be periodically changed so that the analysts do not become familiar with the samples. Intra- and inter-analyst precision may be estimated from blind recounts on reference samples. Inter-analyst precision shall be posted in each laboratory to keep the microscopists informed.

D.6.2.3 Polarized Light Microscopy

Refer to Section D.6.2.1.3.

D.6.3 Other Quality Control Measures

D.6.3.1 Transmission Electron Microscopy

D.6.3.1.1 Water and Wastewater

a) Filter preparations shall be made from all six asbestos types from NIST SRMs 1866 and 1867. These preparations shall have concentrations between 1 and 20 structures (> 10μm) per 0.01 mm^2. One of these preparations shall be analyzed independently at a frequency of 1 per 100 samples analyzed. Results shall be evaluated as verified asbestos analysis in accordance with Turner and Steel (NISTIR 5351).

b) NIST SRM 1876b must be analyzed annually by each analyst. Results shall be evaluated in accordance with limits published for that SRM. Comment: This SRM is not strictly appropriate for waterborne asbestos but analysts can demonstrate general TEM asbestos competence by producing results within the published limits of this (the only recognized TEM counting standard) SRM.

D.6.3.1.2 Air

a) Filter preparations shall be made from all six asbestos types in accordance with Section D.6.3.1.1.a.

b) NIST SRM 1876b must be analyzed annually in accordance with Section D.6.3.1.1.b.

D.6.3.1.3 Solid and Hazardous Waste (Bulk)

All analysts must be able to correctly identify the six regulated asbestos types (chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite). Standards for the six asbestos types listed are available from NIST (SRMs 1866 and 1867). These materials can also be used as identification standards for AEM (Section 3.2.1 Qualitative Analysis, Page 57, EPA/600/R-93/116).

D.6.3.2 Phase Contrast Microscopy

a) Test for Non-Random Fiber Distribution - Blind recounts by the same analyst shall be performed on 10% of the filters counted. A person other than the counter should re-label slides before the second count. A test for type II error (NIOSH 7400, Issue 2, 15 August 1994, Section 13) shall be performed to determine whether a pair of counts by the same analyst on the same slide should be rejected due to non-random fiber distribution. If a pair of counts is rejected by this test, the remaining samples in the set shall be recounted and the new counts shall be tested against first counts. All rejected paired counts shall be discarded. It shall not be necessary to use this statistic on blank recounts.

b) All individuals performing airborne fiber analysis must have taken the NIOSH Fiber Counting Course for sampling and evaluating airborne asbestos dust or an equivalent course.

c) All laboratories shall participate in a national sample testing scheme such as the Proficiency Analytical Testing (PAT) program or the Asbestos Analysts Registry (AAR) program, both sponsored by the American Industrial Hygiene Association (AIHA), or equivalent.

D.6.3.3 Polarized Light Microscopy

a) Friable Materials - Because accuracy cannot be determined by reanalysis of routine field samples, at least 1 out of 100 samples must be a standard or reference sample that has been routinely resubmitted to determine analyst's precision and accuracy. A set of these samples should be accumulated from proficiency testing samples with predetermined weight compositions or from standards generated with weighed quantities of asbestos and other bulk materials (Perkins and Harvey, 1993; Parekh et al., 1992; Webber et al., 1982). At least half of the reference samples submitted for this QC must contain between 1 and 10% asbestos.

b) Non-Friable Materials - At least 1 out of 100 samples must be a verified quantitative standard that has routinely been resubmitted to determine analyst precision and accuracy.

D.6.4 Method Evaluation

In order to ensure the accuracy of reported results, the following procedures shall be in place:

a) Demonstration of Capability – (Refer to Section 5.10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, or method.

b) Performance Audits – (Refer to Section 5.4.2j or 5.5.3.4) The results of such analyses shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.6.5 Asbestos Measurement System Calibration

Refer to methods referenced in the following sections for specific equipment requirements.

D.6.5.1 Transmission Electron Microscopy

AEM (Analytical Electron Microscopy) equipment requirements will not be discussed in this document.

D.6.5.1.1 Water and Wastewater

All calibrations listed below (unless otherwise noted) must be performed under the same analytical conditions used for routine asbestos analysis and must be recorded in a notebook and include date and analyst's signature. Frequencies stated below may be reduced to "before next use" if no samples are analyzed after the last calibration period has expired. Likewise, frequencies may have to be increased following non-routine maintenance or unacceptable calibration performance.

a) Magnification Calibration – Magnification calibration must be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 10,000 and 20,000x. A logbook must be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification. Calibration data must be displayed on control charts that show trends over time. (EPA /600/R-94/134, Method 100.2, Section 10.1)

b) Camera Constant – The camera length of the TEM in the Selected Area Electron Diffraction (SAED) mode must be calibrated before SAED patterns of unknown samples are observed. The diffraction specimen must be at the eucentric position for this calibration. This calibration shall allow accurate (< 10% variation) measurement of layer-line spacings on the medium used for routine measurement, i.e., the phosphor screen or camera film. This must also allow accurate (< 5% variation) measurement of zone axis SAED patterns on permanent media, e.g., film. Calibrations shall be performed monthly to establish the stability of the camera constant (EPA /600/R-94/134, Method 100.2, Section 10.2). Where non-asbestiform minerals may be expected (e.g., winchite, richterite, industrial talc, vermiculite, etc.), an internal camera constant standard such as gold, shall be deposited and measured on each sample to facilitate accurate indexing of zone axis SAED patterns. In such cases, layer line analysis alone shall not be used. Calibration data must be displayed on control charts that show trends over time.

c) Spot Size – The diameter of the smallest beam spot at crossover must be less than 250 nm as calibrated quarterly. Calibration data must be displayed on control charts that show trends over time. (EPA /600/R-94/134, Method 100.2, Section 10.3)

d) Beam Dose - The beam dose shall be calibrated so that beam damage to chrysotile is minimized, specifically so that an electron diffraction pattern from a single fibril ≥ 1 μm in length from a NIST SRM chrysotile sample is stable in the electron beam dose for at least 15 seconds.

e) EDXA System

1) The x-ray energy vs. channel number for the EDXA system shall be calibrated to within 20 eV for at least two peaks between 0.7 keV and 10 keV. One peak shall be from the low end (0.7 keV to 2 keV) and the other peak from the high end (7 keV to 10 keV) of this range. The calibration of the x-ray energy shall be checked prior to each analysis of samples and recalibrated if out of the specified range.

2) The ability of the system to resolve the Na Kα line from the Cu L line shall be confirmed quarterly by obtaining a spectrum from the NIST SRM 1866 crocidolite sample on a copper grid.

3) The k-factors for elements found in asbestos (Na, Mg, Al, Si, Ca, and Fe) relative to Si shall be calibrated semiannually, or anytime the detector geometry may be

altered. NIST SRM 2063a shall be used for Mg, Si, Ca, Fe, while k-factors for Na and Al may be obtained from suitable materials such as albite, kaersutite, or NIST SRM 99a. The k-factors shall be determined to a precision (2s) within 10% relative to the mean value obtained for Mg, Al, Si, Ca, and Fe, and within 20% relative to the mean value obtained for Na. The k-factor relative to Si for Na shall be between 1.0 and 4.0, for Mg and Fe shall be between 1.0 and 2.0, and for Al and Ca shall be between 1.0 and 1.75. The k-factor for Mg relative to Fe shall be 1.5 or less. Calibration data must be displayed on control charts that show trends over time.

4) The detector resolution shall be checked quarterly to ensure a full-width half-maximum resolution of < 175 eV at Mn Kα (5.90 keV). Calibration data must be displayed on control charts that show trends over time.

5) The portions of a grid in a specimen holder for which abnormal x-ray spectra are generated under routine asbestos analysis conditions shall be determined and these areas shall be avoided in asbestos analysis.

6) The sensitivity of the detector for collecting x-rays from small volumes shall be documented quarterly by collecting resolvable Mg and Si peaks from a unit fibril of NIST SRM 1866 chrysotile.

f) Low Temperature Asher - The low temperature asher shall be calibrated quarterly by determining a calibration curve for the weight vs. ashing time of collapsed mixed-cellulose-ester (MCE) filters. Calibration data must be displayed on control charts that show trends over time.

g) Grid Openings - The magnification of the grid opening measurement system shall be calibrated using an appropriate standard at a frequency of 20 openings/20 grids/lot of 1000 or 1 opening/sample. The variation in the calibration measurements (2s) is <5% of the mean calibration value.

D.6.5.1.2 Air

All calibrations must be performed in accordance with Section D.6.5.1.1, with the exception of magnification. Magnification calibration must be done at the fluorescent screen, with the calibration specimen at the eucentric position, at the magnification used for fiber counting, generally 15,000 to 20,000x (AHERA, III.G.1.c). A logbook must be maintained with the dates of the calibration recorded. Calibrations shall be performed monthly to establish the stability of magnification.

D.6.5.1.3 Solid and Hazardous Waste (Bulk)

All calibrations must be performed in accordance with Section D.6.5.1.2.

D.6.5.2 Phase Contrast Microscopy

a) At least once daily, the analyst shall use the telescope ocular (or Bertrand lens, for some microscopes) supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric.

b) The phase-shift detection limit of the microscope shall be checked monthly or after modification or relocation using an HSE/NPL phase-contrast test slide for each analyst/microscope combination (refer to NIOSH 7400, Issue 2, 15 August 1994, Section

10b). This procedure assures that the minimum detectable fiber diameter (< ca. 0.25μm) for this microscope is achieved.

c) Prior to ordering the Walton-Beckett graticule, calibration, in accordance with NIOSH 7400, Issue 2, 15 August 1994, Appendix A, shall be performed to obtain a counting area 100 μm in diameter at the image plane. The diameter, d_c (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule. The field diameter (D) shall be verified (or checked), to a tolerance of 100 μm ± 2 μm, with a stage micrometer upon receipt of the graticule from the manufacturer. When changes (zoom adjustment, disassembly, replacement, etc.) occur in the eyepiece-objective-reticle combination, field diameter must be re-measured (or re-calibrated) to determine field area (mm^2). Re-calibration of field diameter shall also be required when there is a change in interpupillary distance (i.e., change in analyst). Acceptable range for field area shall be 0.00754 mm^2 to 0.00817 mm^2. The actual field area shall be documented and used.

D.6.5.3 Polarized Light Microscopy

a) Microscope Alignment - To accurately measure the required optical properties, a properly aligned polarized light microscope (PLM) shall be utilized. The PLM shall be aligned before each use. (Section 2.2.5.2.3, EPA/600/R-93/116, July 1993)

b) Refractive Index Liquids - Series of n_D = 1.49 through 1.72 in intervals less than or equal to 0.005. Refractive index liquids for dispersion staining, high- dispersion series 1.550, 1.605, 1.680. The accurate measurement of the refractive index (RI) of a substance requires the use of calibrated refractive index liquids. These liquids shall be calibrated at first use and semiannually, or next use, whichever is less frequent, to an accuracy of 0.004, with a temperature accuracy of 2°C using a refractometer or RI glass beads.

D.6.6 Analytical Sensitivity

D.6.6.1 Transmission Electron Microscopy

D.6.6.1.1 Water and Wastewater

An analytical sensitivity of 200,000 fibers per liter (0.2 MFL) is required for each sample analyzed (EPA /600/R-94/134, Method 100.2, Section 1.6). Analytical sensitivity is defined as the waterborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the fraction of the filter sampled and the dilution factor (if applicable).

D.6.6.1.2 Air

An analytical sensitivity of 0.005 structures/mm^2 is required for each sample analyzed. Analytical sensitivity is defined as the airborne concentration represented by the finding of one asbestos structure in the total area of filter examined. This value will depend on the effective surface area of the filter, the filter area analyzed, and the volume of air sampled (AHERA, Table I).

D.6.6.1.3 Solid and Hazardous Waste (Bulk)

a) The range is dependent on the type of bulk material being analyzed. The sensitivity may be as low as 0.0001% depending on the extent to which interfering materials can be removed during the preparation of AEM specimens. (Section 2.5.2 Range, Page 51, EPA/600/R-93/116)

b) There should be an error rate of less than 1% on the qualitative analysis for samples that contain chrysotile, amosite, and crocidolite. A slightly higher error rate may occur for samples that contain anthophyllite, actinolite, and tremolite, as it can be difficult to distinguish among the three types. (Section 3, Page 10, NIST Handbook 150-3, August 1994)

D.6.6.2 Phase Contrast Microscopy

The normal quantitative working range of the test method is 0.04 to 0.5 fiber/cc for a 1000 L air sample. An ideal counting range on the filter shall be 100 to 1300 fibers/mm^2. The limit of detection (LOD) is estimated to be 5.5 fibers per 100 fields or 7 fibers/mm^2. The LOD in fiber/cc will depend on sample volume and quantity of interfering dust but shall be <0.01 fiber/cc for atmospheres free of interferences. (NIOSH 7400, Issue 2, 15 August 1994)

D.6.6.3 Polarized Light Microscopy

The laboratory shall utilize a test method that provides a detection limit that is appropriate and relevant for the intended use of the data. Detection limits shall be determined by the protocol in the test method or applicable regulation.

D.6.7 Data Reduction

D.6.7.1 Transmission Electron Microscopy

D.6.7.1.1 Water and Wastewater

a) The concentration of asbestos in a given sample must be calculated in accordance with EPA /600/R-94/134, Method 100.2, Section 12.1. Refer to Section 5.10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample (EPA /600/R-94/134, Method 100.2, Section 12.2.2).

D.6.7.1.2 Air

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized, e.g., AHERA. Refer to Section 5.10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the upper and lower 95% confidence limits on the mean concentration of asbestos fibers found in the sample.

D.6.7.1.3 Solid and Hazardous Waste (Bulk)

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized (e.g., EPA/600/R-93/116, July 1993). Refer to Section 5.10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties - Proficiency testing for floor tiles analyzed by TEM following careful gravimetric reduction (New York ELAP Certification Manual Item 198.4) has

revealed an interlaboratory standard deviation of approximately 20% for residues containing 70% or more asbestos. Standard deviations range from 20% to 60% for residues with lower asbestos content.

D.6.7.2 Phase Contrast Microscopy

a) Airborne fiber concentration in a given sample must be calculated in accordance with NIOSH 7400, Issue 2, 15 August 1994, Sections 20 and 21. Refer to Section 5.10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Measurement Uncertainties – The laboratory must calculate and report the intra-laboratory and inter-laboratory relative standard deviation with each set of results. (NIOSH 7400, Issue 2, 15 August 1994)

c) Fiber counts above 1300 fibers/mm^2 and fiber counts from samples with >50% of the filter area covered with particulate should be reported as "uncountable" or "probably biased". Other fiber counts outside the 100-1300 fibers/mm^2 range should be reported as having "greater than optimal variability" and as being "probably biased".

D.6.7.3 Polarized Light Microscopy

a) The concentration of asbestos in a given sample must be calculated in accordance with the method utilized (e.g., EPA/600/R-93/116, July 1993). Refer to Section 5.10.6, "Computers and Electronic Data Related Requirements", of this document for additional data reduction requirements.

b) Method Uncertainties - Precision and accuracy must be determined by the individual laboratory for the percent range involved. If point counting and/or visual estimates are used, a table of reasonable expanded errors (refer to EPA/600/R-93/116, July 1993, Table 2-1) should be generated for different concentrations of asbestos.

D.6.8 Quality of Standards and Reagents

D.6.8.1 Transmission Electron Microscopy

a) The quality control program shall establish and maintain provisions for asbestos standards.

   1) Reference standards that are used in an asbestos laboratory shall be obtained from the National Institute of Standards and Technology (NIST), EPA, or suppliers who participate in supplying NIST standards or NIST traceable asbestos. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards shall conform to ANSI N42.22 to assure the quality of their products.

   2) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22-1995, Section 8, Certificates.

b) All reagents used shall be analytical reagent grade or better.

c) The laboratory shall have mineral fibers or data from mineral fibers that will allow differentiating asbestos from at least the following "look-alikes": fibrous talc, sepiolite, wollastonite, attapulgite (palygorskite), halloysite, vermiculite scrolls, antigorite, lizardite,

pyroxenes, hornblende, richterite, winchite, or any other asbestiform minerals that are suspected as being present in the sample.

D.6.8.2 Phase Contrast Microscopy

Standards of known concentration have not been developed for this testing method. Routine workload samples that have been statistically validated and national proficiency testing samples such as PAT and AAR samples available from the AIHA may be utilized as reference samples (refer to Section D.6.2.2b) to standardize the optical system and analyst. All other testing reagents and devices (HSE/NPL test slide and Walton-Beckett Graticule) shall conform to the specifications of the method (refer to NIOSH 7400, Issue 2, 15 August 1994).

D.6.8.3 Polarized Light Microscopy

Refer to Section D.6.8.1.

D.6.9 Constant and Consistent Test Conditions

The laboratory shall establish and adhere to written procedures to minimize the possibility of cross-contamination between samples.